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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

°C	degree Celsius	min	minute
°F	degree Fahrenheit	mm	millimeter
ft	foot	pct	percent
ft^2	square foot	psi	pound per square inch
ft•lbf	foot pound (force)	psi($\sqrt{in}$)	pound per square inch times square root of inches
h	hour	wt pct	weight percent
in	inch		
lb	pound		

FIBER REINFORCEMENT OF SULFUR CONCRETE TO ENHANCE FLEXURAL PROPERTIES

By B. W. Jong,[1] W. C. McBee,[2] K. L. Rasmussen,[3] and T. A. Sullivan[4]

ABSTRACT

The Bureau of Mines studied reinforcement of sulfur concretes using glass, polyester, and aramid fibers. The objective of this research was to determine the feasibility of fiber-reinforcing the sulfur concrete to enhance the flexural properties. Optimum fiber sulfur concrete compositions were formulated with a cement-to-fiber ratio of 9.0:1 for glass-fiber and polyester-fiber sulfur concretes and 12.1:1 for aramid-fiber sulfur concretes.

Test samples were prepared using techniques developed by the Bureau of Mines. Flexural strength values of up to 50 pct of compressive strength values were achieved with fiber additions. With non-fiber-reinforced sulfur concrete, the values ranged up to 20 pct. The impact strength and fracture toughness of sulfur concretes were increased significantly by reinforcement with glass or with aramid fiber and were adversely affected by use of polyester fiber. Strain tolerance was doubled and failure mode was shifted from catastrophic to noncatastrophic type by adding glass fiber. The glass-fiber and aramid-fiber sulfur concretes are resistant to acid corrosion and have proved durable in freeze-thaw cycles. This laboratory and field evaluation has shown that fiber reinforcement of sulfur concrete is feasible and that it enhances the flexural properties.

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INTRODUCTION

The Bureau of Mines initiated a sulfur utilization program in 1972 to develop new uses for sulfur. At that time, large increases in secondary sulfur production through the year 2000 were projected (1).[5] Under the program, the Bureau has developed sulfur composite materials, sulfur-sand-asphalt paving, recycling of spent asphalt paving with sulfur, and sulfur-extended asphalt (2-5). More recently, modified sulfur cements and concretes were developed for use in corrosive industrial environments (6-8).

An industrial evaluation program of sulfur concrete was undertaken with the assistance of The Sulphur Institute and the cooperation of industrial concerns. Test components of sulfur concrete, such as slabs, sump tanks, pump foundations, acid-loading docks, drain ditches, and floors were either precast or constructed in situ and tested in a variety of corrosive environments. The program led to the installation of a total of more than 37,000 ft^2 of sulfur concrete floors. The materials demonstrated their resistance to corrosion in most acid and salt environments in 4 years of exposure (8). It is anticipated that sulfur concrete will be widely used for construction materials in corrosive environments where Portland[6] cement concretes fail.

For concrete paving and some structural applications, flexural strength is more important than compressive strength because a high compressive strength, brittle material with a low flexural strength can fail catastrophically. In the design of concrete paving, flexural strength is a primary factor in determining the necessary thickness. Since thinner concrete floors can be designed using higher flexural strength concrete materials, both materials and costs of construction can be reduced.

The objective of this study was to determine the feasibility of fiber-reinforcing sulfur concrete to increase its flexural properties. These materials could then be used in designs where high impact resistance and thinner sulfur concrete layers were desired. The use of fiber reinforcement of composite materials is not new. Aramid, carbon, and boron fibers have been used in reinforcement of polymers; SiC, Al_2O_3, and tungsten fibers for metal matrix reinforcement; glass for plastics; steel and polyproplyene for Portland cement concretes; and glass and polyester for sulfur concrete (9-13).

Of the fiber materials available, glass, polyester, and aramid fibers were chosen for an initial study on fiber reinforcement of sulfur concretes based on their chemical resistance, lower cost, and commercial availability. The fibers also were available in a chopped form suitable for incorporating in the concrete during the mixing cycle.

MATERIALS

Modified sulfur cement developed by the Bureau of Mines was used. It was prepared by reacting a 5-wt pct mixture of 50 wt pct dicyclopentadiene and 50 wt pct oligomers of cyclopentadiene mixture with 95 wt pct sulfur (6). Dense-graded aggregates were used that contained 55 wt pct of minus 3/8-in crushed quartz, 40 wt pct of minus 1/8-in crushed quartz, and 5 wt pct of minus 200-mesh silica flour. Size distribution of the aggregate is plotted in figure 1, along with a Fuller standard curve for maximum density and minimum voids in mineral aggregate (14). Using a dense-graded aggregate minimizes the cement requirement for the concrete. The size distribution plot of

[5] Underlined numbers in parentheses refer to items in the list of references at the end of this report.

[6] Reference to specific materials does not imply endorsement by the Bureau of Mines.

the aggregate is close to that of the standard curve. Commercially available lengths of chopped glass, polyester, and aramid fibers were used. When a large quantity of fiber was needed in the fabrication of field test specimens, glass roving fibers were used and cut to the required lengths. Physical and mechanical properties of the fibers, as provided by the supplier, are given in table 1.

TABLE 1. - Properties of reinforcing fibers

Fiber	Density, g/cm^3	Young's modulus, 10^6 psi	Tensile strength, 10^3 psi
Glass.....	2.55	10	350
Polyester.	1.38	2	163
Aramid....	1.44	9	400

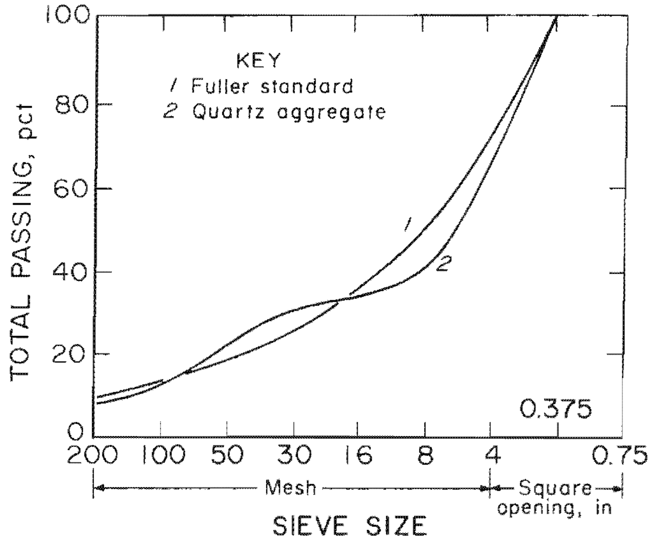

FIGURE 1. - Size distribution of minus 3/8-in aggregate and Fuller maximum-density curve.

MIXTURE DESIGN FOR FIBER CONCRETES

Design objectives were to formulate a workable concrete mixture using aggregate, modified sulfur cement, and fibers to achieve a maximum flexural strength with minimum water absorption. The cement-to-aggregate ratio was held constant and cement-to-fiber ratio was adjusted to the minimum value necessary to achieve a water absorption value for the concrete of less than 0.1 wt pct. This was selected for one of the design criteria for fiber-reinforced concrete because previous studies indicated that the concretes with water absorption values of less than 0.1 wt pct would withstand freeze-thaw cycling in water (7).

Equations 1 and 2 were developed to obtain the cement-to-fiber ratio and to determine the amounts of aggregate and cement to be used. The cement-to-fiber ratio is obtained using equation 1 if the cement-to-aggregate ratio and percent of aggregate, cement, and fiber are known. The aggregate and cement requirements are obtained using equations 1 and 2 by giving the cement-to-aggregate and cement-to-fiber ratios and fiber percent.

$$(C_f/F \text{ ratio})(F) = C_t - (C_a/A \text{ ratio})(A) \quad (1)$$

and

$$A + C_t + F = 100, \quad (2)$$

where A = aggregate, wt pct,

C_t = Total cement, wt pct,

C_a = Cement used for aggregate, wt pct

C_f = Cement used for fiber, wt pct,

F = Fiber, wt pct,

C_a/A ratio = Cement-to-aggregate ratio,

and C_f/F ratio = Cement-to-fiber ratio.

FORMULATION AND TESTING

Test samples, 3- by 3- by 14-in rectangular beams for flexural strength and 3-in-diam by 6-in cylinders for compressive strength, were prepared using glass, polyester, and aramid fibers, modified sulfur cement, and aggregate (15). Fiber lengths of 1/4-, 1/2-, and 1-1/4-in glass; 1/2-, 3/4-, and 1-in polyester; and pulp, 1/4-, 1/2-, and 1-in aramid were used in the study.

For initial mixture design studies, 2 wt pct of 1/2-in glass fiber was selected. Sulfur cement was varied from 31 to 40 wt pct, and aggregate from 58 to 67 wt pct. The aggregate was oven heated to 400° F. The hot aggregate was mixed with sulfur cement (up to 20 wt pct was added as solid, the balance was added as liquid) in an electrically heated (about 250° F) mortar mixer. After the mixture became fluid, the fiber was added and mixing continued for 1 min until the fibers were completely dispersed. Generally, the roving bundles were opened exposing all fibers to the cement during the mix cycle. The mix temperature was maintained at 270° F. The steel beam and cylinder molds were preheated in an oven to 200° F.

The concrete mixture was cast and rodded into the molds in accordance with ASTM Method C31-69, "Making and Curing Concrete Test Specimens in the Field." Samples were left in the molds until they cooled to room temperature. The measurements were determined in accordance with standard ASTM methods for concrete and mineral aggregates (15). Testing for physical, flexural, and compressive strength properties was done in accordance with ASTM Method C642-81, "Specific Gravity, Absorption, and Voids in Hardened Concrete," ASTM Method C78-75, "Flexural Strength of Concrete Using Simple Beam with Third-Point Loading," and ASTM Method C39-80, "Compressive Strength of Cylindrical Concrete Specimens," respectively.

Optimum compositions of 33 wt pct cement and 65 wt pct aggregate were obtained with 2 wt pct of 1/2-in glass fiber. The physical and mechanical properties of the concrete are shown in table 2. The cement-to-fiber ratio of 9.0 was obtained using equation 1. Values of 2 wt pct fiber, 33 wt pct cement, 65 wt pct aggregate, and a cement-to-aggregate ratio of 0.23 were used in the calculation. The cement-to-aggregate ratio of 0.23 was obtained by using a composition of 19

TABLE 2. - Optimum mixture design data

Fiber	Composition, wt pct			C_f/F ratio	Specific gravity	Water absorption, wt pct	Strength, psi		S_f/S_c[1] (100)
	Aggregate	Cement	Fiber				Compressive	Flexural	
Glass:									
1/4-in...	65	33	2	9.0	2.38	0.10	3,995	1,250	31
1/2-in...	73	26	1	9.0	2.26	.10	3,010	1,500	50
	65	33	2	9.0	2.25	.09	4,440	1,870	42
	57	40	3	9.0	2.19	.06	2,115	1,050	50
1-1/4-in.	65	33	2	9.0	2.21	.01	4,535	2,305	51
Polyester:									
1/2-in...	65	33	2	9.0	2.21	.06	2,065	1,075	52
3/4-in...	65	33	2	9.0	2.19	.05	2,985	1,225	41
1-in.....	65	33	2	9.0	2.21	.10	2,998	640	21
Aramid:									
Pulp.....	60	38	2	12.1	2.15	.41	3,235	1,225	38
1/4-in...	60	38	2	12.1	2.17	.01	4,665	1,290	28
1/2-in...	60	38	2	12.1	2.17	.05	4,625	1,210	26
1-in....	60	38	2	12.1	2.15	.03	3,210	1,060	33

[1]Flexural-to-compressive strength ratio.

wt pct cement and 81 wt pct aggregate. This composition was found to be sufficient to coat the aggregate in the concrete mixtures without fiber using 3/8-in dense-graded aggregate (7).

The optimum amounts of aggregate and cement in the mixture design for 1 and 3 wt pct, 1/2-in glass fiber additions were calculated using known cement-to-fiber and cement-to-aggregate ratios. Equations 1 and 2 were used. Values obtained were 26 wt pct cement and 73 wt pct aggregate for 1 wt pct 1/2-in glass fiber, and 40 wt pct cement, and 57 wt pct aggregate for 3 wt pct 1/2-in glass fiber. Values for 2 wt pct 1/4- and 1-1/4-in glass fiber were 33 wt pct cement and 65 wt pct aggregate.

The glass-fiber sulfur concrete test samples were prepared using the compositions obtained. Physical and mechanical properties of the 1 and 3 wt pct of 1/2-in glass fiber and 2 wt pct of 1/4- and 1-1/4-in glass fiber-reinforced sulfur concretes also are shown in table 2.

Concrete mixtures reinforced with 1/2-, 3/4-, and 1-in polyester fibers and pulp, 1/4-, 1/2-, and 1-in aramid fibers were optimized in the same manner. In all cases, 2 wt pct fiber was added. Cement and aggregate compositions were varied using 1/2-in polyester and 1/2-in aramid fibers until the water absorption of the concretes was less than 0.1 wt pct. Optimum compositions of 65 wt pct aggregate and 33 wt pct cement for the 1/2-in polyester and 60 wt pct aggregate and 38 wt pct cement for 1/2-in aramid fibers were obtained. The optimum compositions used for the 1/2-in polyester and aramid fibers were adopted for formulating sulfur concretes with other lengths of polyester and aramid fibers. Physical and mechanical properties of the optimum mix design concretes are summarized in table 2. The cement-to-fiber ratios obtained were 9.0 for polyester concrete and 12.1 for aramid concrete. The results indicate that polyester fibers are similar to glass fibers in the amount of sulfur necessary to coat the fibers; aramid pulp and fibers require more cement to coat the fibers and pulp. The pulp concrete, even at a cement-to-fiber ratio of 12.1, had 0.4 wt pct water absorption. The pulp concrete required so much cement, that its composition was not optimized further. For purpose of comparison, table 3 shows flexural-to-compressive strength ratios and compressive strengths of the glass-, polyester-, and aramid-fiber concrete, the modified concrete, and Portland cement concrete. The results indicate that the fiber, particularly the glass fiber, concretes have a higher range of flexural-to-compressive strength ratios than does Portland cement concrete with similar compressive strength values.

Higher flexural-to-compressive strength ratios were obtained for the fiber-reinforced concretes compared with the modified concretes even though the former had lower compressive strength values. The lower compressive strength of the fiber concretes is probably because the fiber concretes require more sulfur cement (26 to 40 wt pct) than does the modified sulfur concrete (18 to 20 wt pct).

TABLE 3. - Flexural-to-compressive strength ratio and compressive strength of fiber sulfur, sulfur, and Portland cement concretes

Concrete	$S_f/S_c(100)$, pct	Compressive strength, psi	Source of data
Glass sulfur.........	31-51	2,100-4,500	This study.
Polyester............	21-52	2,100-3,000	Do.
Aramid sulfur........	26-38	3,200-4,700	Do.
Modified sulfur......	19-22	4,500-9,000	Refs. 7-8.
Portland cement......	13-18	3,000-4,000	Refs. 16-18.

PREPARATION

The fiber compositions that appeared most promising in the mixture design studies were selected for laboratory and field evaluation. The compositions selected are shown in table 4.

The laboratory test samples were prepared as described in the section on mixture design except that the impact test specimens were formed by extrusion. The following test specimens were used: cylinders, 3 in diam by 6 in, for corrosion tests; beams, 3 by 3 by 14 in, for freeze-thaw durability and fracture toughness tests; beams, 1-1/2 by 1-1/2 by 6 in, for flexural impact tests; 1-1/2-in cubes for compression impact tests; and cylinders, 6 in diam by 12 in, for stress-strain tests. For purposes of comparison, impact test samples of Portland cement concrete were prepared using dense-graded 3/8-in quartz aggregate with six bags of cement per cubic yard at a 0.5 water-to-cement ratio. For preparation of larger field test samples, the aggregate was heated to about 350° F in a propane heated rotary dryer. The aggregates were then mixed with cement and fiber in the mortar mixer. Half of the cement used was liquid at 280° F and half of the cement was in solid flake form. The mix temperature was 270° F. The mixture was poured into 3- by 3-ft by 3-in steel molds or 8- by 8-ft by 5-in wooden molds. The mixtures in the molds were compacted with a vibratory probe and finished with a wooden vibratory screen.

LABORATORY EVALUATION

Test specimens were evaluated in the laboratory for corrosion resistance to immersion in sulfuric acid (H_2SO_4), resistance to freeze-thaw cycling, impact strength, fracture toughness, and stress-strain relationship in compression.

The corrosion resistance of fiber sulfur concretes was evaluated in 20-wt pct sulfuric acid solution which is commonly found in industrial environments. The 3-in-diam by 6-in cylinders of glass-, polyester-, and aramid-fiber sulfur concretes were tested by immersion in the sulfuric acid solution for 1 week and for 2, 3, 6, and 9 months. Three samples of each concrete were removed at the end of each test period. The samples were monitored by determining their compressive strength, weight change, and physical condition. Compressive strengths of the initial samples also were determined. Figure 2 shows plots of the compressive strength over the test period. The materials attained their maximum strength in the sixth month, similar to conventional sulfur concrete aged in air. Compressive strength of the fiber sulfur concretes after 9 months was over 35 pct higher than their initial strength. The samples showed no sign of corrosion and deterioration after 9 months of exposure in the acid solution.

TABLE 4. - Selected compositions for laboratory and field evaluation

Fiber	Compositions, wt pct			Test
	Aggregate	Cement	Fiber	
Glass:				
1/2-in..........	65	33	2	Corrosion, freeze-thaw, impact, fracture toughness, stress-strain, and weathering.
1-1/4-in........	65	33	2	Fracture toughness.
1-in[1]...........	73	26	1	Field impact and corrosion.
1-in............	65	33	2	Do.
Polyester: 3/4-in	65	33	2	Corrosion, freeze-thaw, impact, fracture toughness, and weathering.
Aramid: 1/4-in...	60	38	2	Do.

[1]Obtained by cutting glass rovings.

The freeze-thaw durability of the concretes was performed in accordance with ASTM Method C666-80, "Resistance of Concrete to Rapid Freezing and Thawing, Procedure A, Rapid Freezing and Thawing in Water." The 3- by 3- by 14-in test beams of glass-, polyester-, and aramid-fiber sulfur concretes were cycled in water between -18° and 5° C. The relative dynamic modulus of elasticity of the samples are shown in figure 3. The glass- and aramid-fiber concretes maintained over 40 pct of initial dynamic modulus of elasticy after 300 freeze-thaw cycles. The polyester-fiber concrete failed after 120 freeze-thaw cycles.

The compression impact and flexural impact testing of sulfur concrete, fiber sulfur concrete, and Portland cement concrete specimens was made with an instrumented impact tester. Instrumented impact tests were performed using a drop-weight impact system combined with a data aquisition and analysis system. Data are shown in table 5. Results indicated that the glass-fiber sulfur concretes have two times the compression and flexural impact strengths, and aramid-fiber sulfur concretes have five times the compression and seven times the flexural impact strengths compared with the impact strength of conventional sulfur concrete. The impact strength of polyester-fiber sulfur concretes was less than the impact strength of the sulfur concrete. The compression impact strength of the Portland cement concretes was the same as the compression impact strength of the sulfur concrete. The flexural impact strength of the Portland cement concrete was one-third of the flexural impact strength of the sulfur concrete.

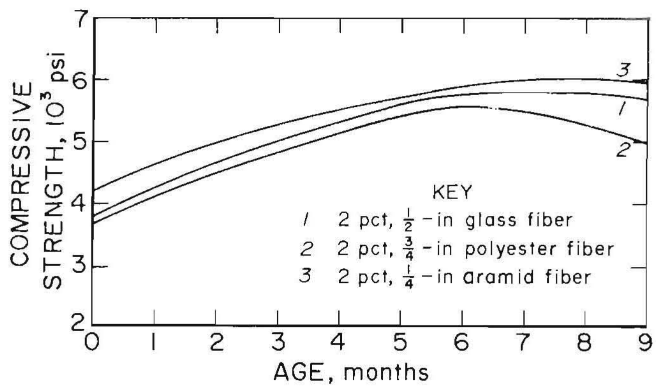

FIGURE 2. - Compressive strength versus age of fiber concrete in 20-wt pct H_2SO_4 solutions.

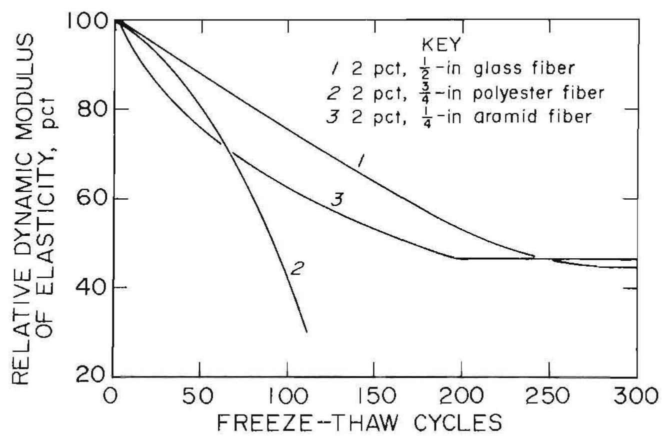

FIGURE 3. - Freeze-thaw durability of fiber concrete.

The fracture toughness of the 3- by 3- by 14-in beams of glass-, polyester-, and aramid-fiber sulfur concretes and conventional sulfur concretes were determined using the same technique as used for

TABLE 5. - Impact strength of concretes

Fiber	Composition, wt pct			Impact strength, ft•lbf	
	Aggregate	Cement	Fiber	Compression	Flexural
None (conventional)....	80	20	0	76	0.6
Glass, 1/2-in..........	65	33	2	148	1.4
Polyester, 3/4-in......	65	33	2	54	NA
Aramid, 1/4-in.........	60	38	2	361	4.0
Portland[1].............	75.3	[2]16.5	0	81	.2

NA Not available. [1]8.2 wt pct H_2O. [2]Portland.

obtaining fracture toughness of Portland cement concretes; a three-point loading flexural test of samples containing a notched flaw (19-20). The following equation was used for calculating the fracture toughness:

$$K_c = \sigma_f Y \sqrt{a}, \quad (3)$$

where K_c = fracture toughness, psi($\sqrt{in}$),

σ_f = flexural strength, psi,

$Y = 1.99 - 2.47(a/b) + 12.97(a/b)^2 - 23.17(a/b)^3 + 24.8(a/b)^4$,

a = notch depth, in,

and b = beam thickness, in.

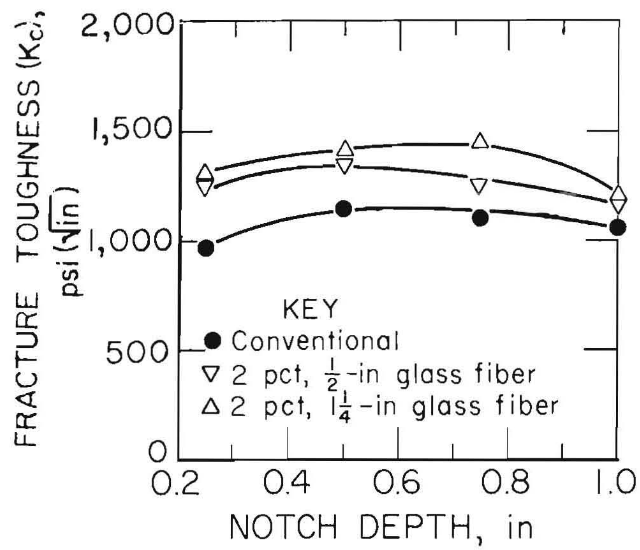

FIGURE 4. - Fracture toughness versus notch depth for conventional and glass-fiber concretes.

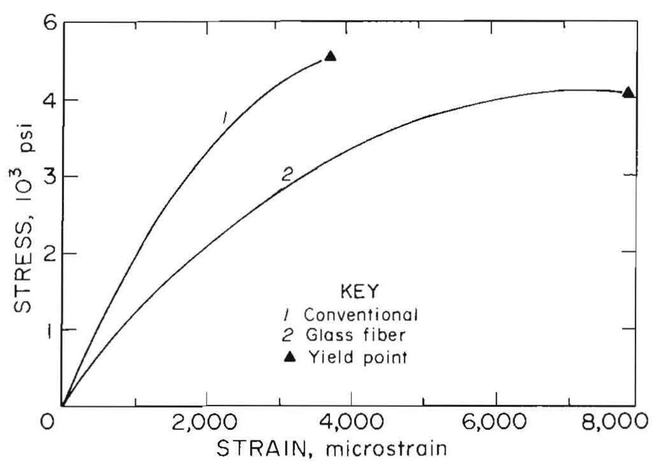

FIGURE 5. - Stress-strain curves for conventional and glass-fiber concretes.

The fracture toughness versus notch depth of the concretes is shown in figure 4. The fracture toughness of the concretes was not greatly affected by the notch depth. The polyester- and aramid-fiber sulfur concretes were therefore notched with a 0.5-in depth and a 0.09-in width for determining their fracture toughness. The fracture toughness of glass-, polyester-, and Aramid-fiber sulfur concrete, and conventional sulfur concrete obtained in this study and fracture toughness of Portland cement concrete obtained from the literature are shown in table 6. Results indicated that glass- and aramid-fiber sulfur concretes have over 10 pct greater fracture toughness than does conventional sulfur concretes, while the fracture toughness of polyester-fiber sulfur concretes and Portland cement concretes was less than that of the sulfur concrete.

The stress-strain curve of glass-fiber sulfur concrete and conventional sulfur concrete was obtained by applying ASTM Method C469-65 "Static Modulus of Elasticity and Poisson's Ratio of Concrete in Compression." The 6-in-diam by 12-in cylinders of glass-fiber sulfur concrete and conventional sulfur concrete were tested using a compressometer. The stress-strain curves for the specimens are shown in figure 5. The glass-fiber sulfur concretes exhibit more plastic yield than do the conventional sulfur concretes. At yield point, the glass-fiber sulfur concrete gave 8,000 microstrain, while conventional sulfur concrete had 3,500 microstrain. Data showed that glass-fiber sulfur concrete has two times more strain tolerance than does conventional sulfur concrete. It also was observed that compared with conventional sulfur contents, glass-fiber sulfur concretes exhibit a longer time to reach failure after yielding. The failure mode of sulfur concrete shifted from brittle-catastrophic to plastic-noncatastrophic type by the addition of glass fiber.

TABLE 6. - Fracture toughness of sulfur, fiber sulfur, and Portland cement concretes

Concrete	Fracture toughness (K_c)		Data source
	psi$\sqrt{in}$	pct[1]	
Sulfur..................	1,130	NAp	This study.
Glass-sulfur, 1/2-in.....	1,350	19	Do.
Glass-sulfur, 1-1/4-in...	1,450	24	Do.
Polyester-sulfur, 3/4-in.	950	-16	Do.
Aramid-sulfur, 1/4-in....	1,220	8	Do.
Portland cement..........	520	-54	Refs. 19-20.

NAp Not applicable.
[1]Strength change compared with strength of sulfur concrete.

FIELD EVALUATION

Test samples of precast fiber sulfur concrete were evaluated for their resistance to weathering, dropping impact, and acid corrosion.

Two precast 3- by 3-ft by 3-in slabs, each of glass-, polyester-, and aramid-fiber sulfur concretes, were installed in the laboratory roadway (fig. 6).

FIGURE 6. - Fiber concrete slabs installed in front of laboratory building.

They were monitored for resistance to weathering and the impact of heavy equipment travel over the slabs. The slabs were cast 2 to 3 in thick, compared with the 4- to 5-in thick sulfur concrete floors generally used in industry. The slabs showed no sign of cracking or disintegration after 9 months of exposure.

A glass-fiber sulfur concrete slab, 8 by 8 ft by 5 in (fig. 7) was cast for testing as part of a battery-dumping pad in a lead-acid battery-recycling plant (21). The fiber sulfur concrete slabs, seven conventional sulfur concrete slabs reinforced with bare or epoxy-coated steel rebar, and one Portland cement concrete slab were installed in the battery-recycling plant as shown in figure 8. An initial test was made using 25-ton truck loads of used batteries dumped onto the slabs from a height of 15 ft to break the batteries to recover their H_2SO_4 and lead contents. No sign of damage to the slabs was observed. The slabs are being visually monitored to assess their long-term durability.

SUMMARY AND CONCLUSIONS

Fiber sulfur concretes were developed and formulated using glass, polyester, and aramid fibers. The compositions of the fiber sulfur concrete were optimized with a cement-to-fiber ratio of 9.0 for glass- and polyester-fiber concretes and 12.1 for aramid-fiber concretes.

Laboratory evaluation of the fiber sulfur concretes showed that flexural strength values were increased from 20 to 50 pct of compressive strength values with fiber addition. The glass- and aramid-fiber concretes were resistant to acid corrosion and were durable to

FIGURE 7. - A glass-fiber concrete pad for use in battery-dumping unit.

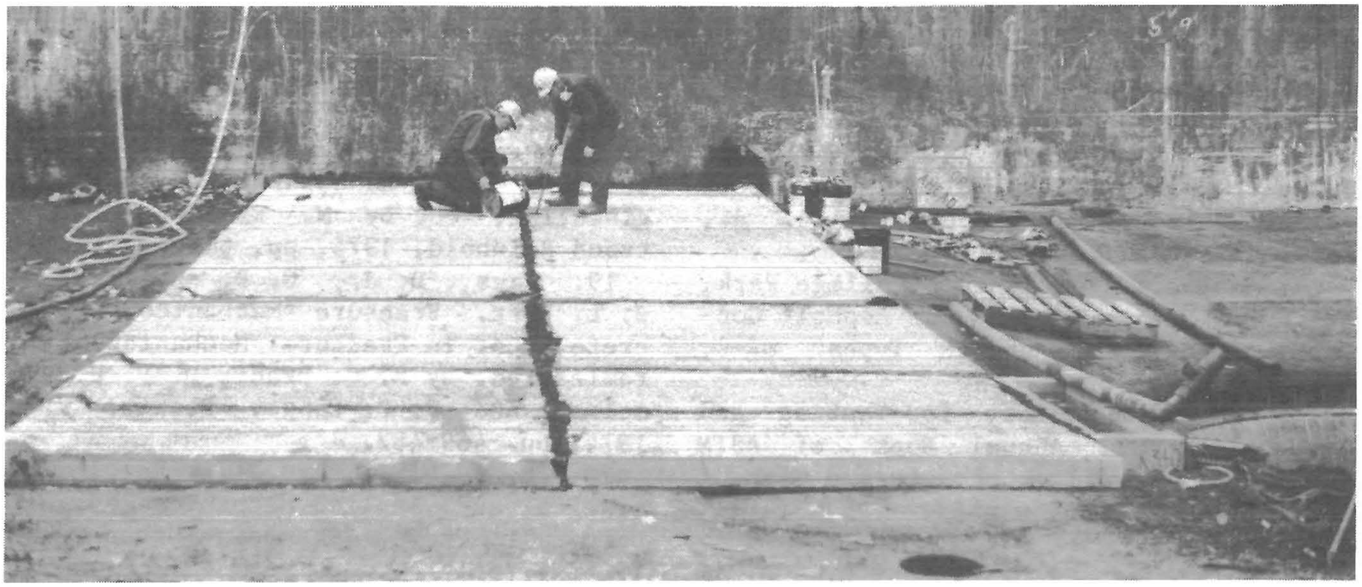

FIGURE 8. - Installed glass-fiber concrete, conventional sulfur concrete, and Portland cement concrete battery-dumping unit.

freeze-thaw cycling. The glass- and aramid-fiber concretes had greater impact strength and fracture toughness than conventional sulfur concrete, while the impact strength and the fracture toughness of the polyester concretes were less than conventional sulfur concrete. Strain tolerance was increased from 3,500 to 8,000 microstrain and the failure mode was shifted from catastrophic to noncatastrophic type with glass fiber addition.

The laboratory and initial field evaluation shows that fiber reinforcement of sulfur concrete is feasible and that it enhances the flexural properties.

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